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IS 14507 (1998): Diphenyl Ether [PCD 18: Natural and Synthetic Fragrance Materials]

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डाईफिनाईल ईथर — विशिष्टि

Indian Standard

DIPHENYL ETHER — SPECIFICATION

ICS 71.080.90

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

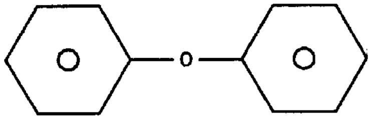
February 1998 **Price Group 2**

Natural and Synthetic Perfumery Materials Sectional Committee, PCD 18

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Diphenyl ether or Diphenyl oxide does not occur in nature. Due to its low cost and stability it is extensively used in various types of fragrances for detergents, soaps and many other household and industrial products for its rose and geranium like odour effect. In conjunction with diphenyl, it is extensively used as a high temperature heat transfer medium (Dowtherm). It is represented by the following structural formula:



(MOLECULAR WEIGHT 170.21)
DIPHENYL ETHER

It is also known as phenyl ether or diphenyl oxide or phenoxybenzene.

In India, diphenyl oxide is produced commercially mainly by two methods. The main method is by heating alkali phenolate with chlorobenzene at elevated temperature in the presence of a catalyst or by passing phenol over a catalyst at 400°C.

In the preparation of this standard, considerable assistance has been derived from the following:

a) Perfumery and Flavouring Synthetics by Paul Z Bedoukian published in 1986;

b) Perfume and Flavour Chemicals, Vol 1, by Steffen Arctander published in 1969; and

c) Givaudan Index (Ed. 2) published in 1961.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DIPHENYL ETHER — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for diphenyl ether.

2 NORMATIVE REFERENCES

The following standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 1984	Sampling (*second revision*)
(Part 3) : 1980	Relative density (*second revision*)
(Part 5) : 1986	Determination of refractive index (*second revision*)
(Part 6) : 1986	Determination of solubility (*second revision*)
(Part 9) : 1980	Determination of ester value after acetylation and free alcohols (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)
2284 : 1988	Methods of olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (*first revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 6597 shall apply.

4 REQUIREMENTS

4.1 Description

4.1.1 The material shall be a clear, nearly colourless liquid, solidifying at or below room temperature. The material shall be free from phenol, moisture and free chlorine and other extraneous matter and impurities.

4.1.2 The material shall also be tested olfactorily and especially for objectionable/undesirable by odours as prescribed under IS 2284.

4.2 Solubility

One gram of the material shall be clearly soluble in 5 parts of ethanol (90 percent by volume) when tested as prescribed under IS 326 (Part 6).

4.3 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Diphenyl Ether

(*Clauses* 4.3 and 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Odour	Strong 'Geranium' leaf like odour, free from phenolic and chloro compounds off-odours	IS 2284
ii)	Relative density at 27/27°C	1.071 - 1.074	IS 326 (Part 3)
iii)	Refractive index at 27/27°C	1.577 2 - 1.578 0	IS 326 (Part 5)
iv)	Congealing point, *Min*, °C	26.7	IS 326 (Part 9)
v)	Freedom from chlorine	Shall pass the test	Annex A
vi)	Freedom from phenol	do	Annex B
vii)	Assay, percent by mass, *Min*	99	Annex C

NOTE — The correction factor for Relative Density and Refractive Index for each degree celsius change in temperature are 0.000 64 and 0.000 38, respectively.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in air-tight and preferably amber-coloured glass, aluminium or tin-plate containers, permitting a minimum of air space, as agreed to between the purchaser and the supplier. The material shall not be packed in galvanized iron or mild steel containers.

5.1.1 The material shall be protected from light and stored in a cool and dry place.

5.2 Marking

Each container so packed shall bear legibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;
c) Batch number and date of manufacture; and
d) Net mass of the material.

5.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1).

6.2 Number of Tests

All the characteristics given under **4** shall be tested on the composite sample.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed under **4.1** to **4.3**. Reference to relevant Indian Standards and Annexes is given in col 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.*(v)]

TEST FOR FREEDOM FROM CHLORINE

A-1 OUTLINE OF THE METHOD

A copper gauge is ignited in the flame with few drops of the sample, if the material is free from halogen, the flame shall show no green colour.

A-2 APPARATUS

A-2.1 Copper Gauge

Strip of 20 mesh and 1.5 cm × 5 cm.

A-2.2 Copper Wire

A-2.3 Bunsen Burner

A-3 PROCEDURE

Wind a strip of 20 mesh copper gauge 1.5 cm long around the end of a copper wire. Heat the gauge in a non-luminous flame of the Bunsen burner until it glows without colouring the flame green. Permit the gauge to cool and re-heat several times until a good coat of oxide has formed. Apply with a medicine dropper, 2 drops of sample to the cooled gauge, ignite, and permit it to burn freely in the air. Again cool the gauge and add 2 more drops of the sample and burn as before. This process is continued until a total of 6 drops has been added and ignited. Then hold the gauge in the outer edge of the Bunsen flame, adjusted to a height of about 4 cm. If the material is free from halogen, the flame should show no green colour.

ANNEX B

[*Table* 1, *Sl No.*(vi)]

TEST FOR FREEDOM FROM PHENOL

B-1 OUTLINE OF THE METHOD

The aqueous filterate of the material is treated with ferric chloride solution, a greenish, purplish or bluish colour indicates the presence of phenol or cresol.

B-2 REAGENTS

B-2.1 Ferric Chloride Solution

10 percent.

B-3 PROCEDURE

Shake vigorously 1 ml of the sample with 10-20 ml of distilled water in a glass-stoppered test tube. Allow the liquids to separate and filter through filter paper moistened with distilled water. To the aqueous filtrate, collected in a test tube, add 2-3 drops of a 10 percent solution of ferric chloride. A greenish, purplish or bluish colour indicates the presence of cresol or phenol.

ANNEX C

[*Table* 1, *Sl No.*(vii)]

DETERMINATION OF PURITY

C-1 OUTLINE OF THE METHOD

A sample of the material is dissolved in a suitable solvent (for example, cyclohexane, hexane, or petroleum ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column. The detector signals, on transmission to the recorder plots the chart. From the specific area under various peaks corresponding to the specific constituents, the quantities of different constituents are determined.

C-2 APPARATUS

Any suitable gas chromatograph and column capable of being operated under conditions suitable for resolving the individual constituent into distinct peaks may be used. The typical chromatogram for diphenyl with the following chromatographic conditions is shown in Fig. 1:

Sample	Diphenyl ether
Column	
Material	Stainless steel
Length	3 m
I.D.	0.318 cm
Stationary phase and solid support	15 percent FFAP by mass on chromosorb WHP 80-100 mesh
Carrier gas	Nitrogen
Conditions	
Column temperature (isothermal)	200°C
Injection port temperature	230°C
Detector	
Type	FID
Temperature	250°C

C-3 PROCEDURE

The gas chromatograph is prepared for GLC analysis of diphenyl ether under the conditions indicated in **C-2** or are set using pure samples of diphenyl ether. After stabilization of the desired temperature of the column, injection port and detector, 0.2 to 1.0 microlitre of the sample is injected with microlitre hypodermic syringe. On the chromatogram obtained, all peaks of interest should be of suitable dimensions and that except in case of attenuation, none should exceed 90 percent of all the available recorder paper width. If required, sample is re-injected to get a better chromatogram.

C-4 CALCULATION

Peak areas are calculated either by the most commonly used triangular method or automated integration. When an electronic integrator is used, concentrations of the constituents on the basis of the peak areas on chromatogram are automatically calculated and presented as a printout. For specific constituents, internal standard method is employed for higher accuracy.

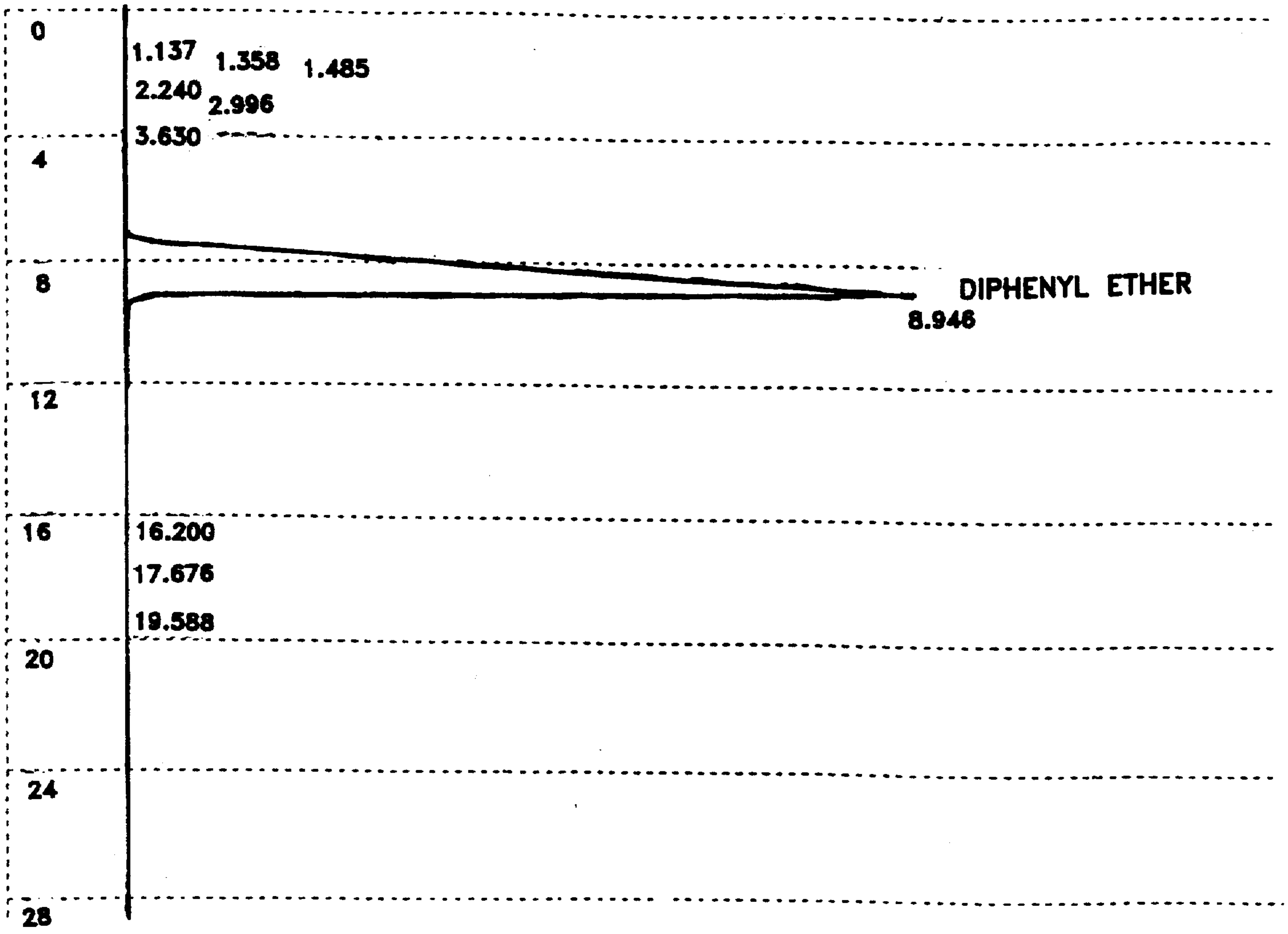


FIG. 1 TYPICAL CHROMATOGRAM OF DIPHENYL ETHER

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Amendments Issued Since Publication

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